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AD904414

CHEMICAL VAPOR DEPOSITION

OF IR MATERIALS FOR RECONNAISSANCE

B. A. diBenedetto

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Raytheon Company

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TECHNICAL REPORT AFAL-TR-72-309

SEPTEMBER 1972

Air Force Avionics Laboratory

Air Force Systems Command

Wright-Patterson Air Force Base, Ohio 45433

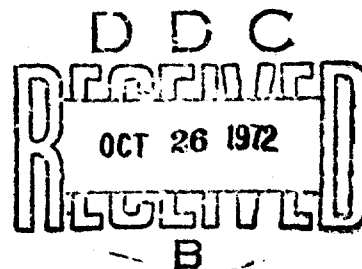# CHEMICAL VAPOR DEPOSITION

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B. A. diBenedetto

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FOREWORD

This report was prepared by Raytheon Company, Research Division, Waltham, Mass., under Contract No. F33615-71-C-1775, Project No. 6102, Task 01, entitled, "Chemical Vapor Deposition of IR Materials for Reconnaissance." The work was administered under the direction of the Air Force Avionics Laboratory, Wright-Patterson Air Force Base, Ohio. Mr. C. T. Ennis (TEL) was task engineer.

At Raytheon, the investigation was carried on in the Advanced Materials Department of the Research Division. Dr. J. Pappis is Department Manager, and Mr. B. A. diBenedetto, principal investigator.

This is the Final Technical Report for Contract F33615-71-C-1775. It covers the period June, 1971 to June, 1972. The report was given the Raytheon internal number S-1468.

This report was submitted by the authors August, 1972.

This technical report has been reviewed and is approved.

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ABSTRACT

The significance of this research and development program to the Air Force is the demonstrated feasibility of fabricating large infrared transmitting windows of zinc sulfide with good physical characteristics by the chemical vapor deposition (CVD) process. Plates approximately 1 ft. $\times$ 2 ft. with good transmission between 8 and 12 μm were made and delivered as window blanks. Furthermore, the imaging properties of CVD ZnS, particularly in the visible end of the spectrum, were significantly improved in comparison to material fabricated during an earlier program. It was also shown that it may be possible to extend the useful transmission range of zinc sulfide by fabricating a solid solution with zinc selenide.

Material fabricated during this program had a transmittance in excess of 60 percent from 6 to 12.5 μm, a flexural strength of 15,000 psi, and a use limit above 200° C. The potential for improving the optical quality of the material through better control of the process and/or annealing the material after deposition was also demonstrated.

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INTRODUCTION

Reconnaissance and weapon delivery applications require infrared windows that can be fabricated into large sizes and various geometric shapes. For example, FLIR type applications require large size IR windows with good physical properties to withstand severe environmental conditions. For the past decade considerable effort has been devoted toward the development of suitable windows. Window blanks have been formed by special glass casting, hot pressing and crystal growth techniques, but none of the processes have yielded totally satisfactory windows. Another process that was initially explored under contract F33615-70-C-1577 and found to yield good optical quality material is the chemical vapor deposition process. This process is not inherently size or shape limited, and it has the potential of fabricating good optical quality polycrystalline infrared transmitting material. The Air Force Avionics Laboratory, recognizing the potential of this process, continued the effort initiated under the original contract to further develop zinc sulfide and to scale the process to fabricate large aperture infrared windows.

Raytheon Company's approach for this program has been to: 1) Make exploratory depositions of zinc sulfide (4 × 4 × 1/8 in. in size) to optimize the deposition parameters in order to minimize optical scattering (particularly in the visible), 2) Once adequate control of the process was achieved 6 × 12 × 1/4 in. and 13.5 × 24 × 1/2 in. plates were fabricated, 3) Determine the properties from the various runs including the larger plates, and deliver the latter materials to the Air Force Avionics Laboratory for further evaluation. Thus an iterative type program was conducted whereby changes were made in each run based on the evaluation of the previous run.

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SECTION II
THE CHEMICAL VAPOR DEPOSITION PROCESS

The chemical vapor deposition (CVD) process offers many advantages over conventional techniques for preparing infrared transmitting materials. Perhaps the two most significant advantages are that the resulting material is very pure, thus eliminating IR absorptions due to impurities and that the deposits are usually very dense, and thus light scattering due to pores is minimized. Furthermore, the process is not inherently size limited and it has the potential of fabricating polycrystalline infrared windows in large sizes and various shapes.

The chemical vapor deposition process can be summarized as follows: Volatile compounds of the elements comprising the material to be deposited are reacted at a surface whose temperature allows the compound to decor pose or react to form a solid, adherent coating. If the coating thickness is heavy enough a monolithic free-standing plate is obtained. The volatile byproducts of the reaction are pumped away, flushed away in a stream of carrier gas, removed by reaction with a mass of suitable material in the system, or regenerated or used by reaction with a reservoir of a suitable raw material.

Chemical vapor deposition processes can be used to form the most refractory substances at temperatures where their vapor pressure is negligible. The properties of the deposited materials can be significantly and controllably altered by the co-deposition of alloying atoms. Depending on the relative concentration of the reactants, either solid solutions or two-phase composites can be formed. Crystallite orientation and size distribution can be controlled by proper manipulation of the deposition parameters. Composites with alternating layers of two or more different materials can be prepared by cycling the composition of the vapors from which the materials are deposited.

Two general types of systems, static and dynamic, can be used for chemical vapor deposition. The static system is a closed system, in which the reactants and products are sealed in a chamber. Well-known examples are the quartz-iodine incandescent lamp and the hydro-thermal bomb for the deposition of synthetic quartz. In the dynamic system, on the other hand, fresh reactants are continuously metered into the deposition chamber, and the spent vapors are continuously removed, usually by pumping. The reactive gases are fed into the furnace through a gas-metering system. The substrate upon which the deposit occurs is maintained at an appropriate temperature by means of a heater that is inductively or resistively heated. Most vapor depositions are made at pressures on the order of one-hundredth of an atmosphere, although a much higher or lower pressure can be employed.

Our experiments have shown that the dynamic system yields good results in the deposition of zinc and cadmium sulfide and zinc selenide, and is preferable because it offers certain advantages. Chief among these is the depletion of reactants and the accumulation of waste materials which are major problems in the static system, whereas the dynamic system allows the addition and removal of materials during deposition thus minimizing these problems. In order to obtain a deposit, the temperature of the substrate chamber and the vapor source are usually more critical and interdependent in the static system than in the dynamic system. The static system, in general, offers less flexibility in the deposition parameters than the dynamic system, since vapor transport is controlled by temperature gradients rather than pressure gradients and mass flow. In addition, the static system is often more susceptible to vapor phase nucleation and particle growth near the substrate; to reduce this effect the partial pressures of the reactive vapors must be low, resulting in low deposition rates. The reactive vapor concentrations are also limited by the equilibrium constants of regenerative reactions and by the fact that partial pressures of the regenerative vapor cannot (for safety reasons) usually greatly exceed one atmosphere. Finally, outgassing of deposition

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chamber and substrate are of greater importance in the static system than in the dynamic system.

Two general techniques can be employed in vapor deposition. These are: 1) Conventional chemical vapor deposition where the vapor source temperature is lower than the substrate temperature; 2) Transport chemical vapor deposition, where the vapor source temperature is greater than the substrate temperature.

In conventional chemical vapor deposition, the thermodynamics and kinetics of the chemical reactions are such that formation of the solid product is favored at the higher temperatures, whereas the volatile reactants tend to be formed or are stable at the lower temperatures.

In chemical transport deposition, on the other hand, the thermodynamics and kinetics of the chemical reactions are such that formation of the solid product is favored at the lower temperatures, whereas the volatile reactants are formed at the higher temperatures.

The initial experiments at Raytheon employed chemical transport deposition in a dynamic system using HCl to transport zinc sulfide. It was soon discovered that conventional chemical vapor deposition in a dynamic system yielded superior results, and this method was used exclusively to fulfill the objectives of this program.

SECTION III
CONVENTIONAL CVD OF ZINC SULFIDE - EXPLORATORY

During the preceding contract a process had been established which yielded theoretically dense ZnS. The material, however, exhibited considerable scatter particularly in the visible end of the spectrum. There were also problems with cracking of large plates of this material because of the mandrel design. Considerable additional knowledge was needed regarding details of the Zn and H_2S reaction process, as well as improved mandrel and setup design to enable the fabrication of large plates of acceptable optical quality material.

The exploratory runs of this program were made in a manner that allowed us to evaluate the effect of variations in process parameters on properties. Forty-one process evaluation runs were made and they are outlined in Table I. The following sections describe the pertinent details of these exploratory runs and how material properties were affected by changes in process parameters.

Initially, considerable time was spent on setting up furnaces and accessory equipment that could safely handle hydrogen sulfide, a toxic gas, hold a large supply of zinc, and maintain it at a constant temperature for continuous evaporation purposes. A good deal of time was also spent setting up a permanent scrubbing system to neutralize any excess hydrogen sulfide. The excess hydrogen sulfide was passed through a KOH solution being sprayed into two chemical reaction towers placed in series (Fig. 1). This system was designed such that it could handle the anticipated loads which might result from incomplete reaction even for the large plates to be prepared in the scale-up section of this contract. The scrubbing system has a detector solution at the end of the chain to indicate whether any H_2S has not reacted in the towers prior to exhausting to the atmosphere. Several trial runs were made to check out the

TABLE I

ZINC SULFIDE DEPOSITION RUNS

Run No.	Retort Temp (°C)	Mandrel Temp (°C)	Pressure (torr)	Zn Pickup Rate (gm/hr)	H_2S Flow (lpm)
ZS-38	600	650	40	127	1.5
ZS-39	600	750	40	127	1.5
ZS-40	600	700	40	136	1.5
ZS-41	600	650	20	206	1.5
ZS-42	600	650	40	154	1.5
ZS-43	600	650	80	90	1.5
ZS-44	600	600	40	127	1.5
ZS-45	600	550	40	155	1.5
ZS-46	600	550	40	137	1.0
ZS-47	600	500	40	121	1.5
ZS-48	550	500	15	129	1.5
ZS-49	550	550	40	97	1.5
ZS-50	600	550	40	213	1.5
ZS-51	550	550	40	74	1.5
ZS-52	600	550	40	83	1.5
ZS-53	600	750	40	233	1.5
ZS-54	600	550	80	26	1.5
ZS-55	600	550	20	129	1.5
ZS-56	600	550	20	109	1.5
ZS-57	620	550	20	245	3.0
ZS-58	600	550	40	199	3.0
ZS-59	560	550	40	124	3.0
ZS-60	560	550	40	88	3.0
ZS-61	600	700	40	199	1.5
ZS-62	600	700	40	103	0.8
ZS-63	600	550	40	34	1.5

Table I Cont'd)

ZINC SULFIDE-SELENIDE DEPOSITION RUNS

Run No.	Zn Retort Temp (° C)	Mandrel Temp (° C)	Pressure (torr)	Zn Pickup Rate (gm/hr)	H_2S Flow (lpm)	H_2Se Flow (lpm)
ZS-64	600	650	40	198	0.9	0.1
ZS-65	600	750	40	124	0.9	0.1
ZS-66	600	550	40	---	0.9	0.1
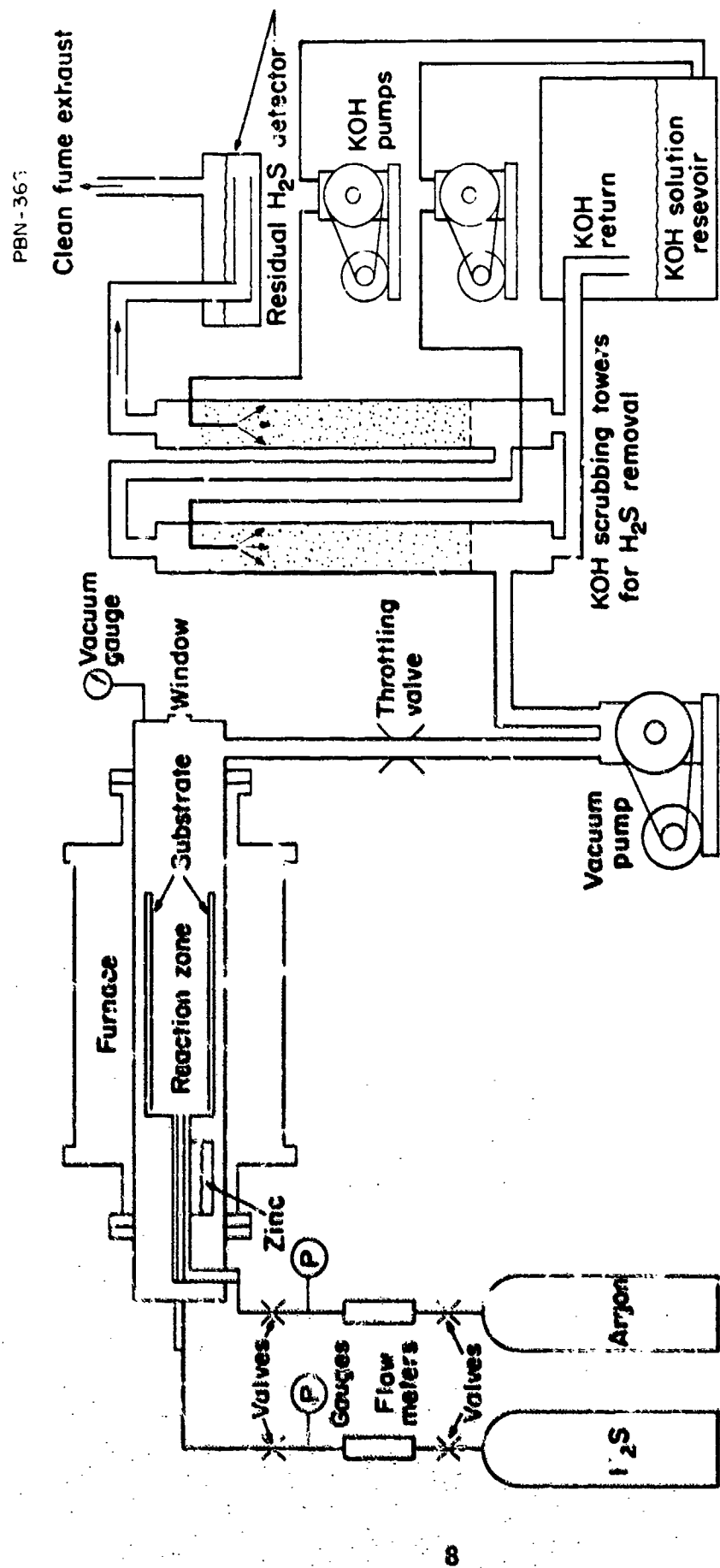
Fig. 1 Schematic of ZnS CVD system

efficiency and completeness of the scrubbing action which took place in the towers. When positive results from these tests were attained, the initial process runs were begun. Prior to the runs the temperature profile of the furnace was adjusted so that the deposition zone was controlled to $\pm$ 10° C.

The first parameter evaluated in this series of runs was the deposition temperature (i.e., the temperature of the mandrel). The temperature was varied between 500° and 750° C. The adjustment of the mandrel temperature was made while holding all other process parameters constant. Later, other process parameters were adjusted along with the mandrel temperature; they are discussed below. A marked difference was noted in the material over this range of deposition temperature. Material deposited at temperatures below 600° C exhibits a fine grain size and is noticeably better in its visible imaging quality than material deposited at higher temperatures. The higher temperature deposited material (> 660° C) also is of larger grain size. The other major difference in the materials prepared under these conditions is that the low temperature material contained an optical absorption at approximately 6 μm (see Figs. 2, 3, and 4).

A second parameter which was adjusted systematically was the furnace pressure, or the pressure which was maintained within the mandrel during deposition. This pressure was adjusted in various runs from a low of 10 torr to a high of 80 torr. No major material differences were noted as a result of this change within this range. Furnace pressure does, however, have an effect on other parameters, such as zinc evaporation, which strongly controls the quality of the material. Therefore, there were some differences in the material which were indirect results of variations in furnace pressure. It would be expected that the pressure in the reaction zone will have a bearing on the dissociation of the zinc sulfide that is formed, and hence cause a major difference in the deposition rate and the quality of the material. This effect was not a dominant factors over the range of pressures covered in this study.
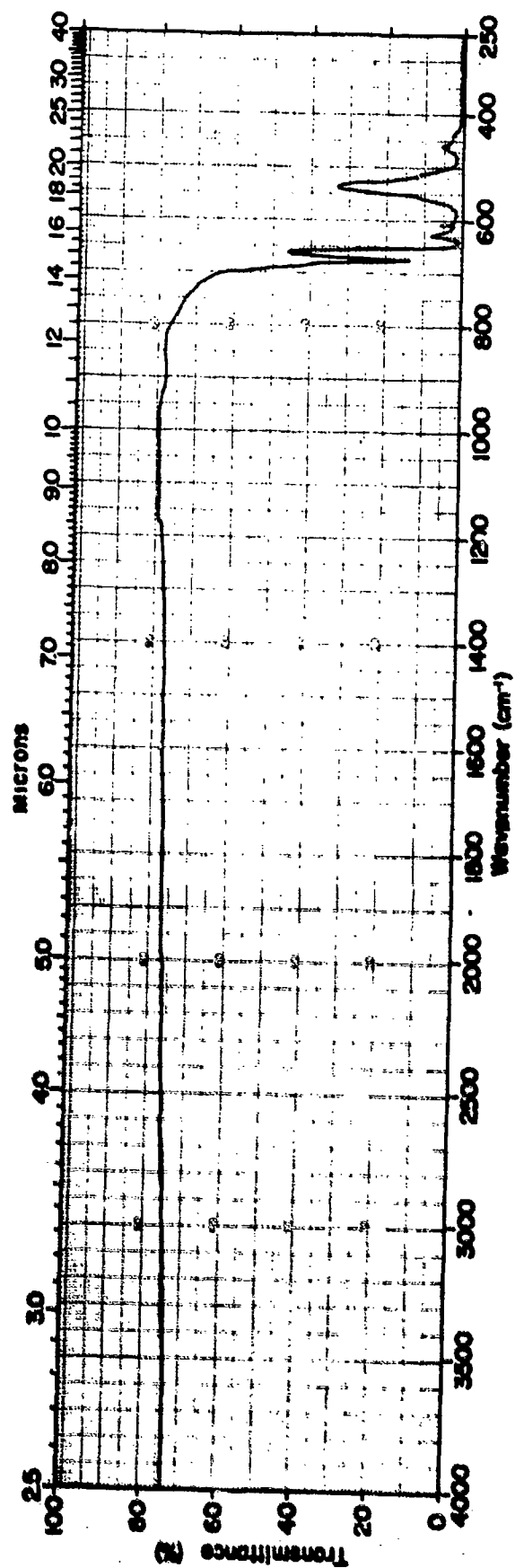
Fig. 2.   IR Transmission for ZS-39, Deposition Temperature
750° C, Sample Thickness 0. 025 in.

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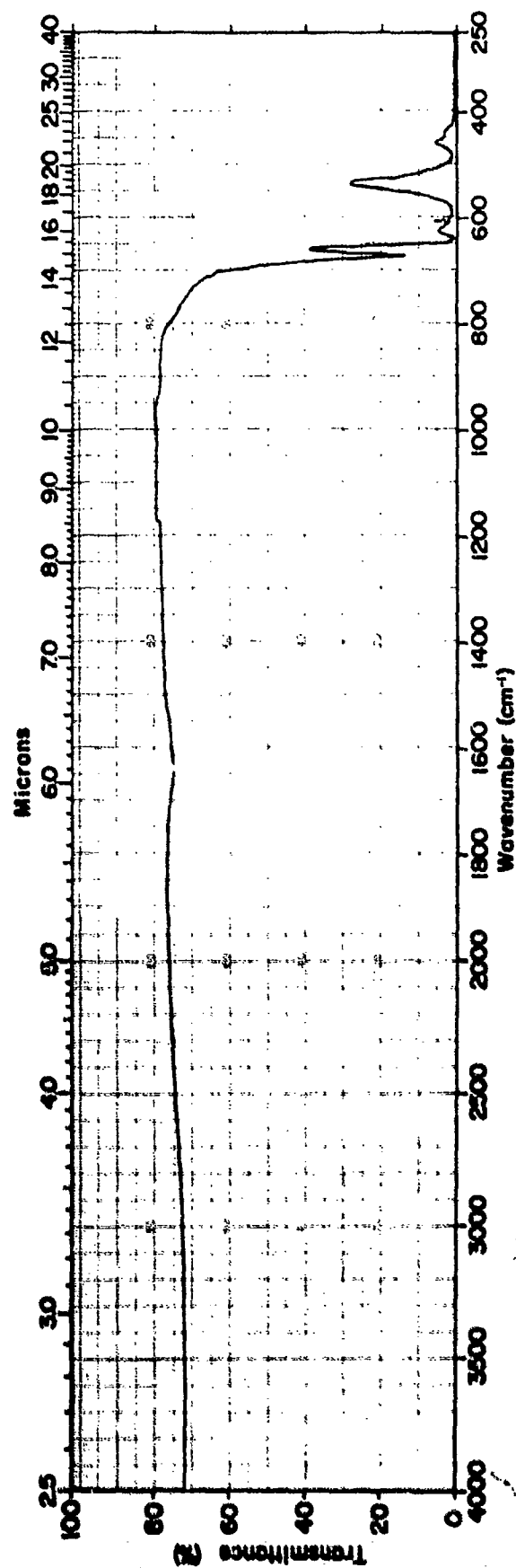
Fig. 3.   IR Transmission for ZS-38, Deposition Temperature 650° C, Sample Thickness 0. 029 in.
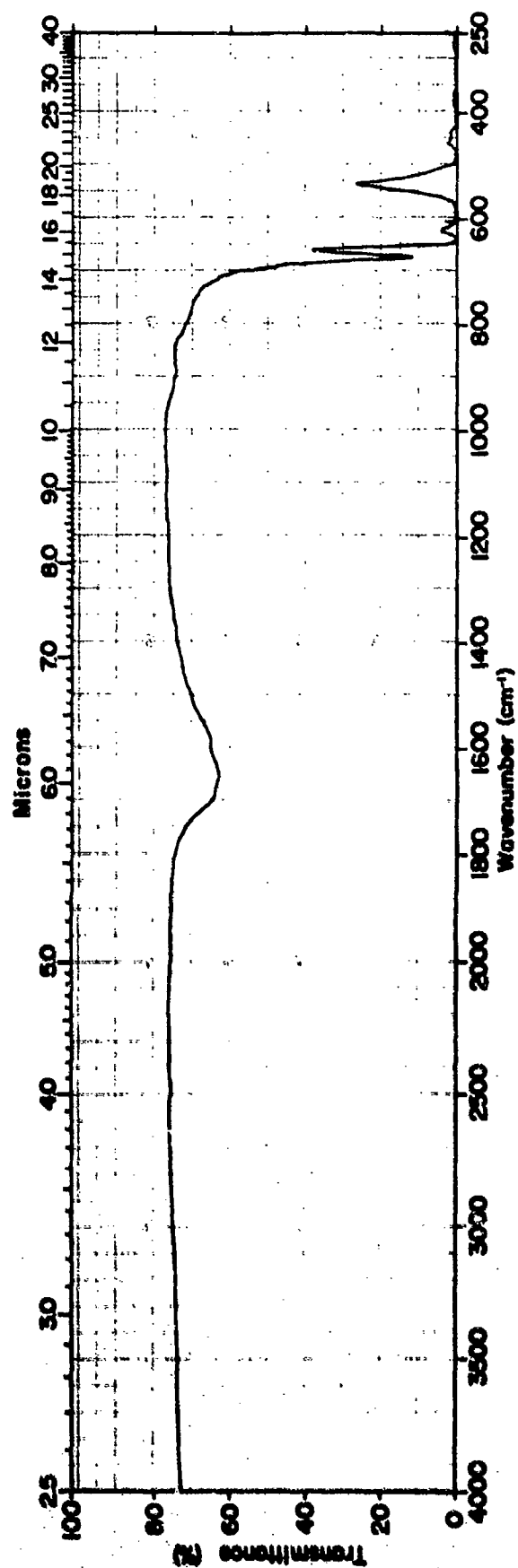
Fig. 4. IR Transmission for ZS-45, Deposition Temperature 550° C, Sample Thickness 0.030 in.

A third factor which was considered during the process runs was the stoichiometry of the material and the stoichiometry of the gases during deposition. The molar ratio of the reactant gases H_2S/Zn was varied from 0.2 to 14.0. This was done by either lowering or raising the H_2S flow or by changing the zinc vapor flow by adjusting the temperature of the zinc retort and/or furnace pressure. Analysis of this series of runs indicated that the lower the H_2S/Zn ratio the better the optical imaging quality of the material. For this reason considerable effort was placed on adjusting and monitoring this ratio to determine if an optimum ratio could be established. The results of these runs indicated that the lower the ratio the better the imaging quality of the material. However, a practical limit was reached as to how much zinc could be handled by the existing equipment. During runs where exceedingly high zinc flow rates were used, droplets of zinc were commonly found imbedded in the matrix of the material. When these runs were carried out for long periods of time, clogging of the exhaust system with zinc dust became a common problem.

Attention was also given to the adjustment of the partial pressures of the reacting species by diluting with inert gas or by increasing the gas velocity through the mandrel area. This was done in order to reduce gas phase reactions and to limit the reaction and growth to the mandrel wall. In terms of the optical quality of the CVD zinc sulfide, no improvement was seen as a function of reacting gas concentrations. However, it had an adverse effect of reducing the deposition rate.

Another factor that was explored in attempting to reduce and/or eliminate scattering was the reactivity of the H_2S and zinc vapor. If the reaction is too rapid gas phase reactions might occur and large nucleii could be incorporated into the deposit to cause scattering. For this reason 0.1 lpm of HCl vapor was added to run ZS-77 to determine if the rate of reaction could be altered or the mobility of the reacting species could be

enhanced. The results of this run were negative; i.e., the imaging resolution was degraded in the visible.

Pure zinc sulfide can exist in both the cubic (sphalerite) and the hexagonal (wurtzite) forms, and the coexistence of both these forms can be a source of optical scattering centers. Small additions of zinc selenide to the zinc sulfide reportedly insure the deposition of only the cubic phase of zinc sulfide. If the scattering centers are related to the mixture of cubic and hexagonal stacking in the pure zinc sulfide material, the addition of hydrogen selenide to the hydrogen sulfide reactant gas could possibly reduce or eliminate the source of scattering. Three runs were carried out at various mandrel temperatures in order to quickly assess the feasibility of this approach. In the runs that were made no improvement was noted. In retrospect, however, it is believed that improvements were not seen since optimum growth conditions in terms of other equally important process conditions, such as H_2S/Zn molar ratio, were not correct.

Another parameter which was adjusted in order to insure growth at the mandrel wall was the temperature of reacting gases. Optimum reaction kinetics can be controlled by cooling or heating the reacting gases. If the gases are preheated they will react immediately and tend to increase gas phase reactions. If the gases are too cool they will react incompletely and possibly lead to the growth of nonstoichiometric materials. Several runs were made in which the hydrogen sulfide gas was preheated before it entered the reaction chamber. In run ZS-72 the hydrogen sulfide gas was deliberately cooled before it entered the reaction chamber. None of these changes appeared to significantly alter the imaging quality of the zinc sulfide that was deposited.

All of the process runs were made in furnaces that contain graphitic components. It was speculated that the 6 μm absorption band and/or the scattering centers could be caused by the presence of CS_2 or some other carbonaceous species in the system. To test this hypothesis run

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ZS-67 was made in a quartz furnace completely free of graphitic components. The deposition temperature was 550° C where the 6 μm absorption band is pronounced. All other process conditions were similar to those of all 550° C depositions. The result of this run were equivalent to runs made in graphite component furnaces, indicating that carbonaceous species are not the cause of absorption or imaging problems.

One final variable that was explored was mandrel material. Three materials were investigated and all three appear to be satisfactory. These are graphite coated with colloidal graphite, graphite coated with pyrolytic carbon, and optically polished fused silica. The material that was utilized more than any of the others was graphite coated with colloidal graphite. The colloidal graphite acts as a film on which the deposited material does not adhere. Equivalent results were obtained on graphite coated with pyrolytic carbon but unless a stable coating is initially deposited, bubbling of the coating can occur that in turn can cause a nonuniform thickness profile. Polished silica also was successful; however its initial expense, particularly in large size plates, negated its use.

SECTION IV
SCALE-UP STUDIES FOR ZINC SULFIDE

The final objective of this program was to fabricate and deliver large plates of CVD zinc sulfide to the Air Force Avionics Laboratory. The blank sizes that were our goal were nominally sized at $9 \times 10 \times 0.370$ in., $10 \times 10 \times 0.370$ in., and $14 \times 17 \times 0.500$ in. The problems encountered in scaling the process were somewhat different from the process study runs. Procedures had to be established so that deposition rates could be maintained constant over large areas. All cracking had to be eliminated so that blanks of the desired size could be deposited and delivered. In addition, it was necessary to maintain constant deposition conditions over the long periods of time in order to deposit plates of the required thickness. As a consequence of these requirements extreme care was needed in the setup and operation of each run.

Scale-up runs were carried out in two steps. For the initial step the size of the mandrel was increased from the standard $4 \times 4 \times 9$ in. size to an intermediate $7 \times 7 \times 16$ in. mandrel. Six runs were carried out using this size mandrel. The primary purpose of these intermediate scale-up runs was to determine the effect of higher mass flows on deposition rate, thickness distribution, and optical imaging quality. During these runs and the final scale-up runs as well, the best process conditions as determined in the process run phase of this study, were used. These runs are summarized in Table II.

Several parameters were adjusted during these runs in an effort to fabricate high quality optical material. One obvious adjustment was an increase in the total gas input in order to maintain an adequate deposition rate for the larger mandrel areas. Also, the run time was extended in order to increase the thickness over that required in the normal process runs. Deposition times up to 76 hours were used in order to attain deposits

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TABLE II

ZINC SULFIDE DEPOSITION RUNS - SCALE-UP

Run No.	Retort Temp (°C)	Mandrel Temp (°C)	Pressure (torr)	Zn Pickup Rate (gm/hr)	H_2S Flow (lpm)
ZS-70	600	640	40	38	3.0
ZS-73	630	650	40	500	1.5
ZS-75A	630	650	40	285	1.0
ZS-81	630	650	40	620	1.0
ZS-86	640	650	10/60	160	1.0
ZS-87	630/640	650	40	420	1.0
ZS-89	640	650	45	730	1.0
ZS-90	640	650	45/60	630	2.0
ZS-91	640	650	50	690	2.0
ZS-92	630	650	43	475	2.0
ZS-93	640	650	50	488	2.0
ZS-94	640	650	40	726	2.0
ZS-95	640	650	40	781	2.0
ZS-96	640	650	40	564	2.0

with thicknesses up to 0.375 in. Also, due to the much larger quantities of zinc being consumed from the zinc retort, larger retorts had to be designed and care had to be taken so that constant evaporating temperatures and pressures could be maintained.

The first positive result of these runs that was determined almost immediately was that the optical imaging quality of the material was quite uniform over the entire area of the plates. Slightly more scatter was noted in the lower part of each plate; however this was not considered to be a serious problem. The thickness profile in these intermediate scale-up runs was extremely good. As noted from the results of run ZS-73, the thickness variation for a plate 7 X 16 in. in size at a thickness of 0.1 in. is less than 10 percent.

One new problem which developed in these intermediate scale-up runs was the clogging of the exhaust end of the furnace. The unreacted gases which were being passed over the mandrel tended to deposit out in the exhaust section, and over a long period of time tended to decrease the exhaust area and lead to pressurization and other related problems. This problem dictated the redesign of the exhaust end of the furnace so that clogging could be reduced to a minimum for additional scale-up runs.

For the final scale-up runs plates 16 X 24 X 0.5 in. in size were desired. Mandrels used for this phase of the program were 24 X 16 X 3 in. in size and two CVD ZnS plates could be yielded from each run. Nine runs were carried out using these large mandrels in a furnace which was capable of handling these larger sizes. These runs are also summarized in Table II.

In these final scale-up runs the thickness profile immediately became a problem primarily because of the nonsymmetry of the mandrel. This led to additional modifications which had to be made at the gas inlet area of the furnace. The initial runs made, ZS-87, ZS-89, ZS-90, and

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ZS-92, were trial runs to evaluate the new inlet design and the new exhaust area designs.

Though cracking was evident in these early runs, complete 16 X 24 in. plates were easily taken from all but one of the five final runs. All cracking that was evidenced, originated from areas where the zinc sulfide adhered to the mandrel and thereby loaded the plate in tension. Mandrels were designed so that no adherence was possible and only improperly assembled mandrels had this adherence problem.

The optical quality of these large ZnS plates was judged to be good in accordance with the criteria that was utilized to judge the process runs. In some instances minor amounts of zinc were observed in the plates. For FLIR type applications, however, this defect was not considered to be a serious detriment. The optical quality of these runs, in general, was not considered to be as good as the best of the process runs. Some minor variations were noted in the optical quality of the material in the axial, transverse, and thickness directions. Window blanks were cut and ground from runs ZS-91, ZS-93, and ZS-95, and submitted to the Air Force Avionics Laboratory for further evaluation. Optical and mechanical properties from these runs are given in Fig. 5 and Table III.

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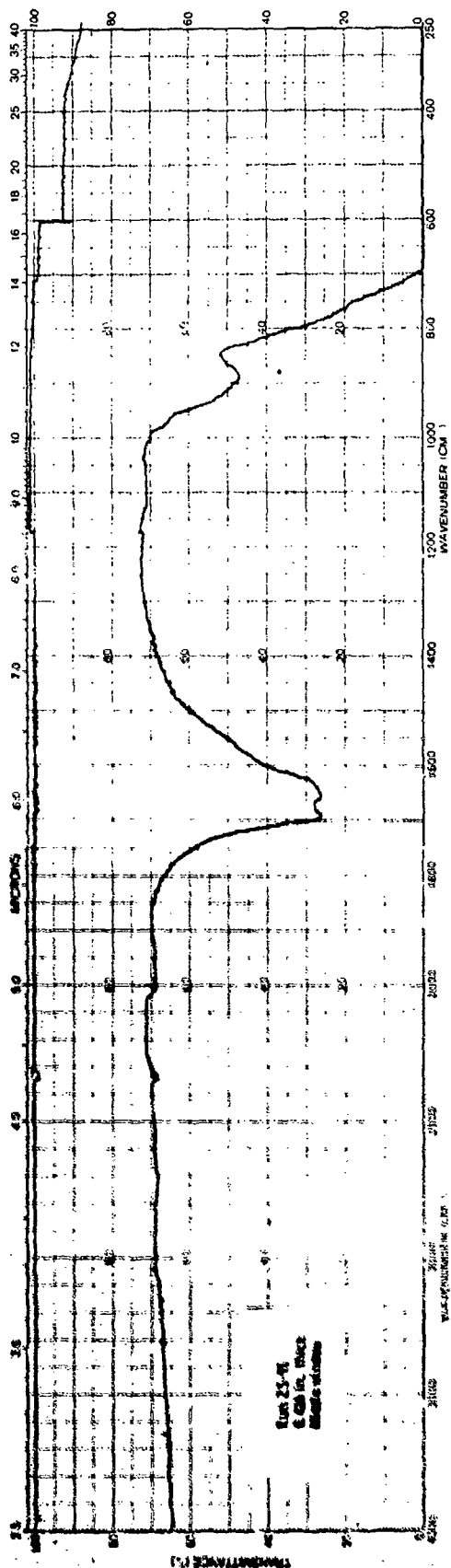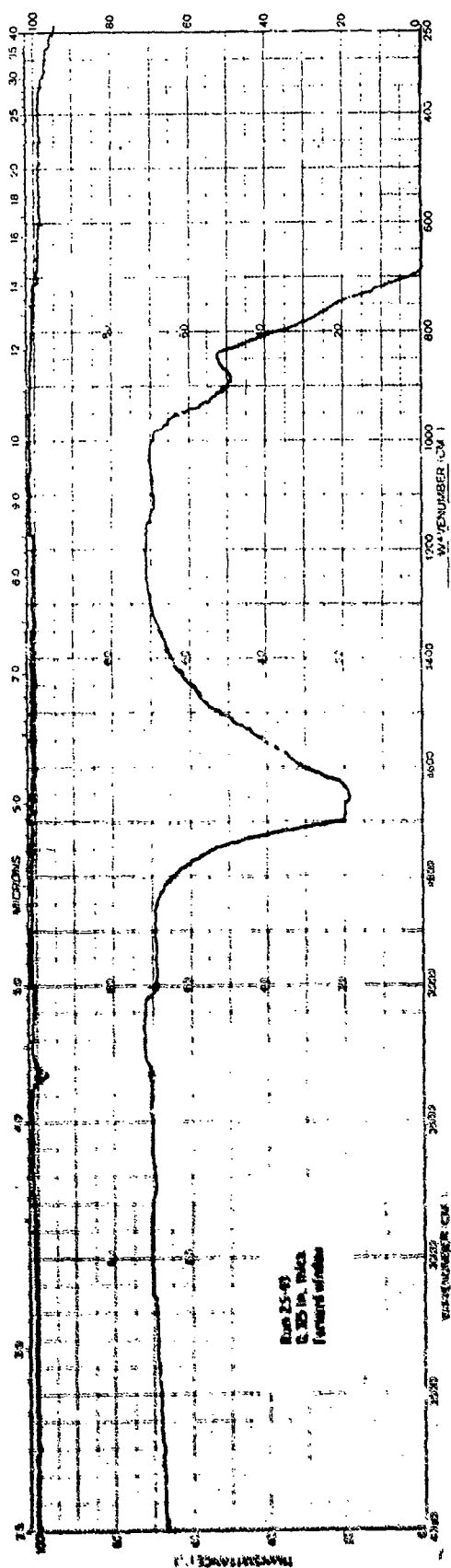
Fig. 5. IR Transmission for Submitted Plates

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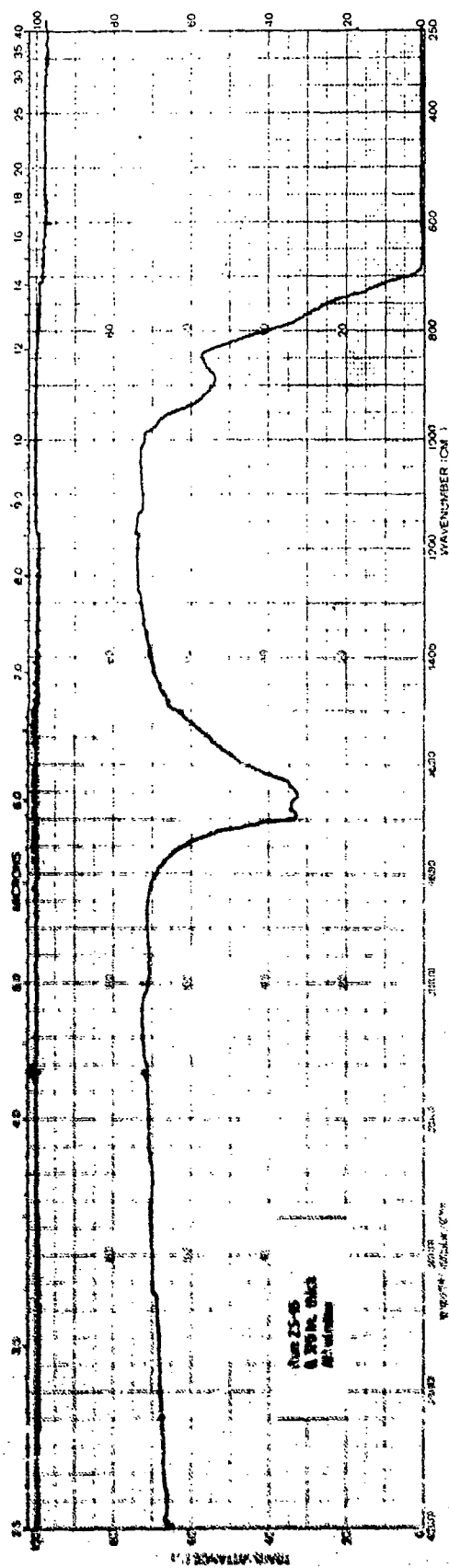
TABLE III

MECHANICAL PROPERTIES OF PROTOTYPE CVD ZnS

Four-Point Loading Span = 2" Load distributed over 1"			Three-Point Loading Span = 1" Load applied at center of beam		
Run No.	Sample No.	Flex. Str. (psi)	Run No.	Sample No.	Flex. Str. (psi)
91	1	9,680	91	1	13,300
	2	10,840		2	12,480
	3	9,700		3	14,920
	4	10,670		4	10,310
	5	11,210		5	14,120
	6	10,850		6	15,700
	7	9,020		7	11,520
	Avg.(7)	10,280 ± 740		8	10,400
93	1	12,970		9	10,650
	2	10,050		Avg.(9)	12,600 ± 1900
	3	8,910	93	1	10,350
	Avg.(3)	10,640 ± 1700		2	11,840
95	1	9,060		3	13,360
	2	13,140		4	16,140
	3	9,450		5	15,910
	4	12,940		6	12,820
	5	10,880		Avg.(6)	13,400 ± 2080
	6	10,040	95	1	17,270
	7	14,280		2	12,140
	8	9,840		3	16,900
	9	12,040		4	12,940
	Avg.(9)	11,300 ± 1750		5	14,440
				6	14,310
				7	15,640
				8	13,160
				Avg.(8)	14,600 ± 1750

SECTION V
PROPERTIES AND CHARACTERIZATION

All the deposits discussed in the previous two sections of this report were subjected to various evaluations and the results of these evaluations provided the direction to be followed in subsequent process runs, as well as the scale-up runs. Properties measured were in-line transmission (0.5 to 20 μm), optical scattering, X-ray lattice constants, mechanical properties, and spectrographic analysis for impurities. In addition, a limited study was carried out to determine the effect of annealing CVD ZnS in various atmospheres.

During the exploratory phase of the program the primary concern of the program was to determine the cause of scattering centers in material and to devise means of minimizing and/or eliminating them. Many samples were observed under a polarizing microscope and in a scanning electron microscope to determine if discrete scattering centers, such as pores or inclusions, could be observed and identified. No centers could be isolated indicating they were very small. In addition, the stoichiometry of material that was deposited was investigated to determine if stoichiometry could be correlated with scattering and/or the 6 micrometer absorption band. Attempts to identify free carriers by resistivity measurements gave very little insight to the problem mainly because the as-deposited material exhibited a high resistivity ($\sim 2 \times 10^{11}$ ohm-cm).

1. OPTICAL TRANSMISSION

Infrared transmission of samples from all runs was measured on a Perkin Elmer 457 grating spectrometer. Samples 1 $\times$ 1/2 $\times$ 0.030 - 0.600 in. in thickness were evaluated. These samples generally showed greater than 70 percent in-line transmission from 2.5 μm to 11 μm and they often displayed a slight upward slope from 2.5 μm to the long

wavelength cutoff. A portion of the slope is caused by surface scatter and a portion is caused by bulk scatter. A common occurrence in the low temperature deposited material was an absorption band in the 6 μm region. Figures 2, 3, and 4 represent the spread in optical transmission properties observed in the process runs. There is no 6 μm absorption band in Fig. 2 which is typical of the high temperature deposits ($\sim$750° C). The absorption band becomes more pronounced as seen in Figs. 3 and 4 as the deposition temperature is decreased to 650° and 550° C respectively. Differences in the imaging resolution in the visible is quite noticeable in these samples. The clearest image is exhibited by material deposited at 550° and 650° C even though the in-line transmission curves are not significantly different. Figure 6 shows a series of in-line transmission curves from run ZS-91 at thicknesses up to 0.600 in. The difference in the percent transmission is mainly due to bulk scattering. The intrinsic absorption at the long wavelength cutoff is more pronounced in the thicker samples. In addition, the 6 μm absorption band is more pronounced; however, this absorption can be minimized as discussed in the section on annealing studies.

The optical transmission in the visible range of the spectrum was measured on selected samples using a Cary Model 14 spectrometer. The same samples used in the infrared measurements were also used for the visible spectrum evaluation. Figures 7 and 8 show the complete transmission curve from 0.2 to 40 μm for two samples from runs ZS-89 and ZS-91. The ZS-91 sample exhibited a slight absorption band at 6 μm, while run ZS-89 does not. This 6 μm absorption band is almost always accompanied by a yellow coloration of the material. In the absence of the 6 μm absorption band the material appears colorless. Comparison of the visible cutoff in Figs. 7 and 8 shows that the colorless material (ZS-89) has a steeper slope than ZS-91. The apparent absorption in the blue region for sample ZS-91 is what gives the material its characteristic yellow color.
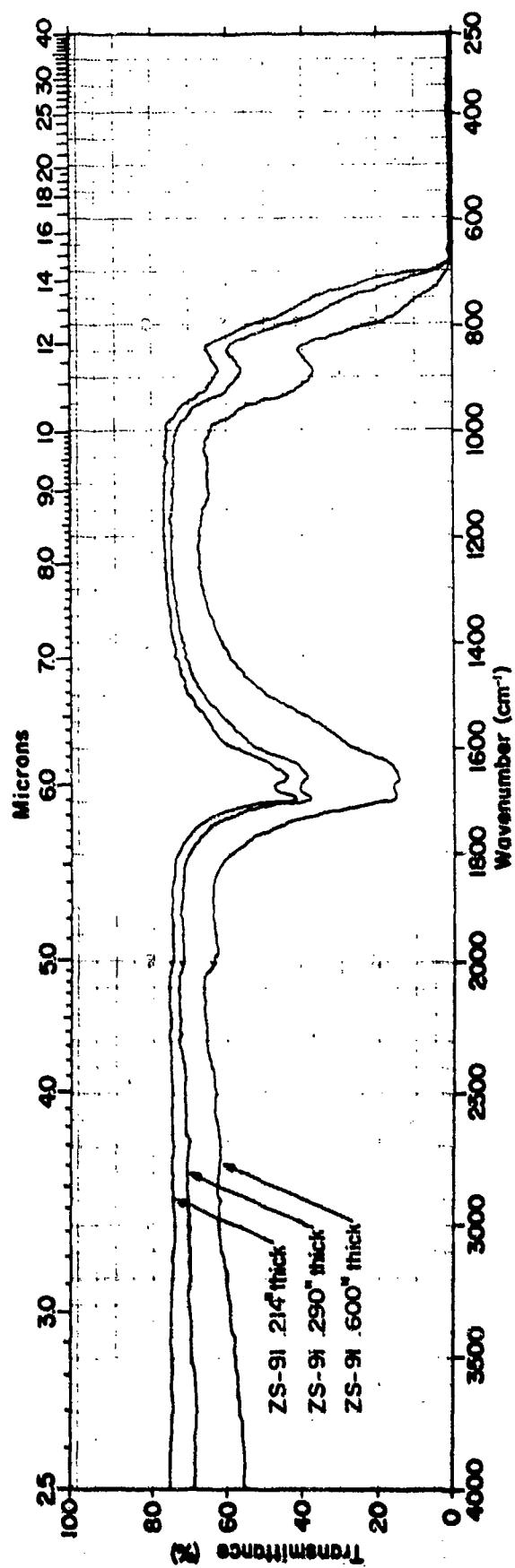
Fig. 6.   IR Transmission for Three Thicknesses.
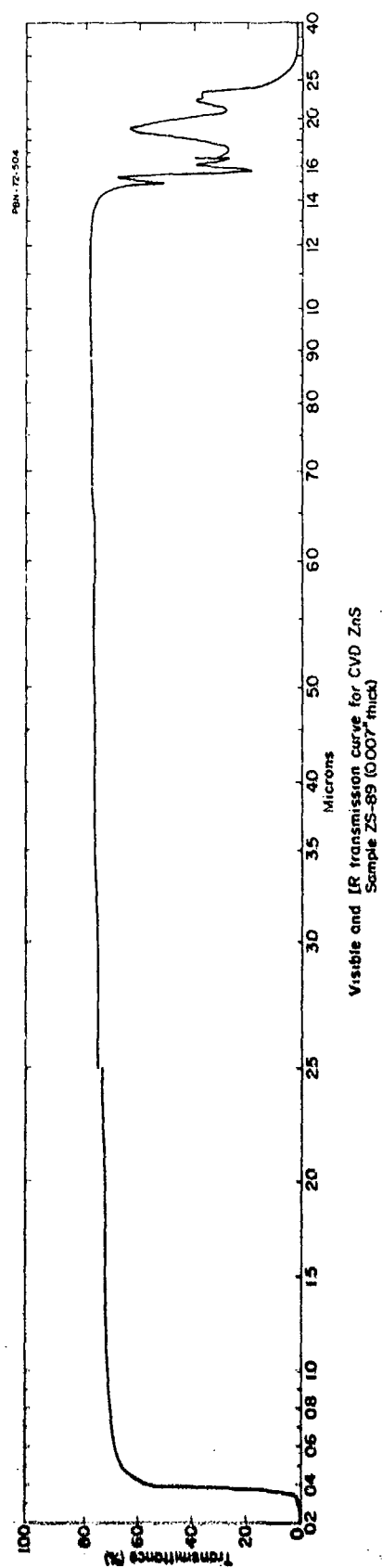
Fig. 7.   Visible and IR Transmission for ZS-89.

PBN-72-504

Visible and IR transmission curve for CVD ZnS
Sample ZS-89 (0007" thick)

Visible and IR transmission curve for CVD ZnS
Sample ZS-91 (0010" thick)
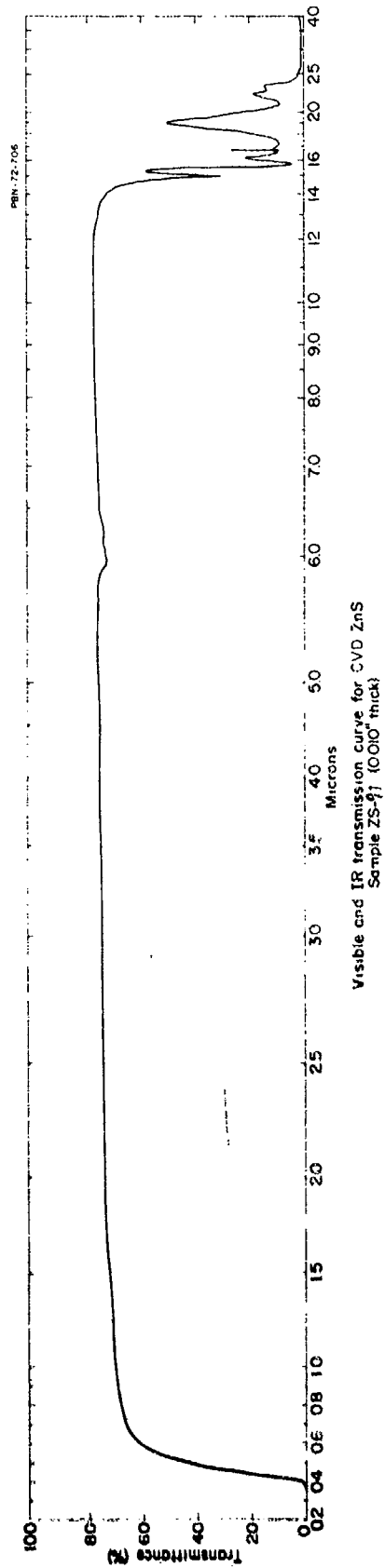
Fig. 8.   Visible and IR Transmission for ZS-91.

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2.0 SCATTERING MEASUREMENTS

It is obvious from the transmission curves, such as the lower curve in Fig. 6 that there is considerable scattering in some of the zinc sulfide CVD material deposited to date. The inclining slope of the transmission curve as a function of wavelength is interpreted to be mainly due to scattering loss. However, it is unclear how much of the scatter is low angle or high angle scattering. The best means of detecting low angle scatter is by visual imaging through polished samples. If an image can be seen through the material in the visible then the amount of scattering at longer wavelengths would be expected to be less. For practically all of the high temperature deposits no images could be identified through even 0.030 in. thick samples. However, extremely clear images could be seen through samples deposited at lower temperatures and in fact thin material was deposited in which slope between 2.5 and 11 μm was essentially zero.

Figure 9 is a photograph taken of a heated electric light bulb filament through ZnS plates. To qualitatively determine the imaging quality of the material, thin (0.030 in.) polished specimens fabricated under different process conditions were placed in front of the filament, and the clarity of the filament was observed. Note the clarity of the filament diminishes from left to right. In the upper section of the figure the intensity of the filament was kept constant. In the lower section of the figure an attempt was made to define the filament even further by filtering out some of the light source. It is evident from either the upper or lower sequences that the sample ZS-45 has the least amoung of low-angle scatter of all specimens shown. Our best estimate of the scattering loss is 100 μrads or less. There still appears to be some high-angle scattering as evidenced by the halo around the filament. Sample ZS-45 was deposited at 550° C.

The high-angle scatter indicated in Fig. 9 obscures a well-defined image which is apparent once the high-angle scatter is filtered out. The

No
filter

With
filter

No   ZS-45   ZS-38   ZS-39
sample
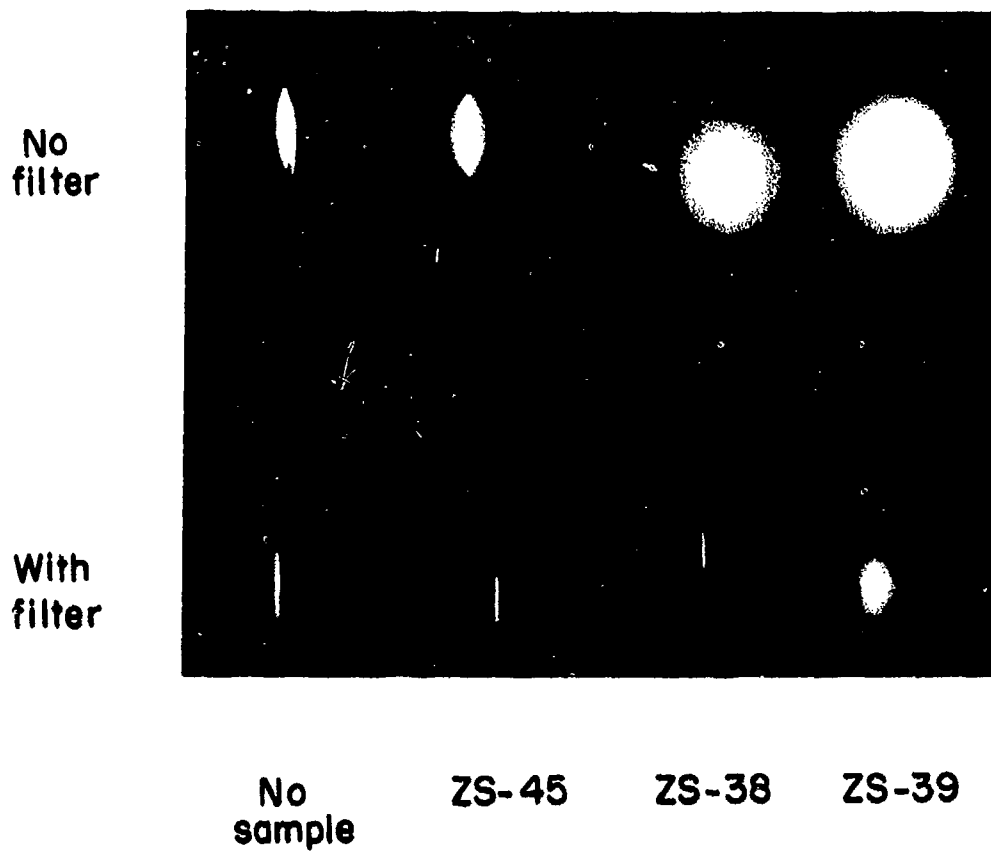
Fig. 9.   Qualitative Measurements of Scatter Loss.

question obviously arose: was high-angle scatter also a problem in the 8 - 12 μm range, the wavelengths of interest in this program? In order to determine if this was the case, high-angle scatter measurements were made at 3.39 μm on material which obviously scattered in the visible (ZS-70), material which was extremely clear in the visible (ZS-74), and a sample of CVD zinc selenide which was of good optical quality. For this purpose a chopped laser beam was passed through the samples as shown schematically in Fig. 10. A detector was mounted so it could scan 90° from the incident beam and the relative scatter intensity could be detected. Figure 11 shows the scattered intensity per thickness of these samples vs the angle from the transmitted beam. The relatively low total scatter intensity (as compared to the intensity of the transmitted beam) and the minor difference in scattering between samples corroborates the conclusions drawn from the transmission curves, i.e., that the scattering beyond the visible end of the spectrum is not appreciable for the present quality CVD zinc sulfide.

3.0 ANNEALING STUDY

Annealing experiments were carried out to substantiate some of our conjectures on the structure and stoichiometry of CVD ZnS. It is known that zinc sulfide can exist in both the cubic (sphalerite) and hexagonal (wurtzite) forms, and that there is no general agreement under which conditions each of these modifications can be formed. We believe one of the factors that controls whether the hexagonal or cubic phases are deposited in the CVD process is the partial pressures of zinc and sulfur. We also feel that the 6 μm absorption we observe in the low temperature deposits is either an impurity phenomena or a stoichiometry problem. The scattering observed in the high temperature deposits could be associated with the presence of the hexagonal phase in which small pores (< 1000 Å) are entrapped as the material is deposited. The annealing experiments were thus designed to answer some of the above conjectures.
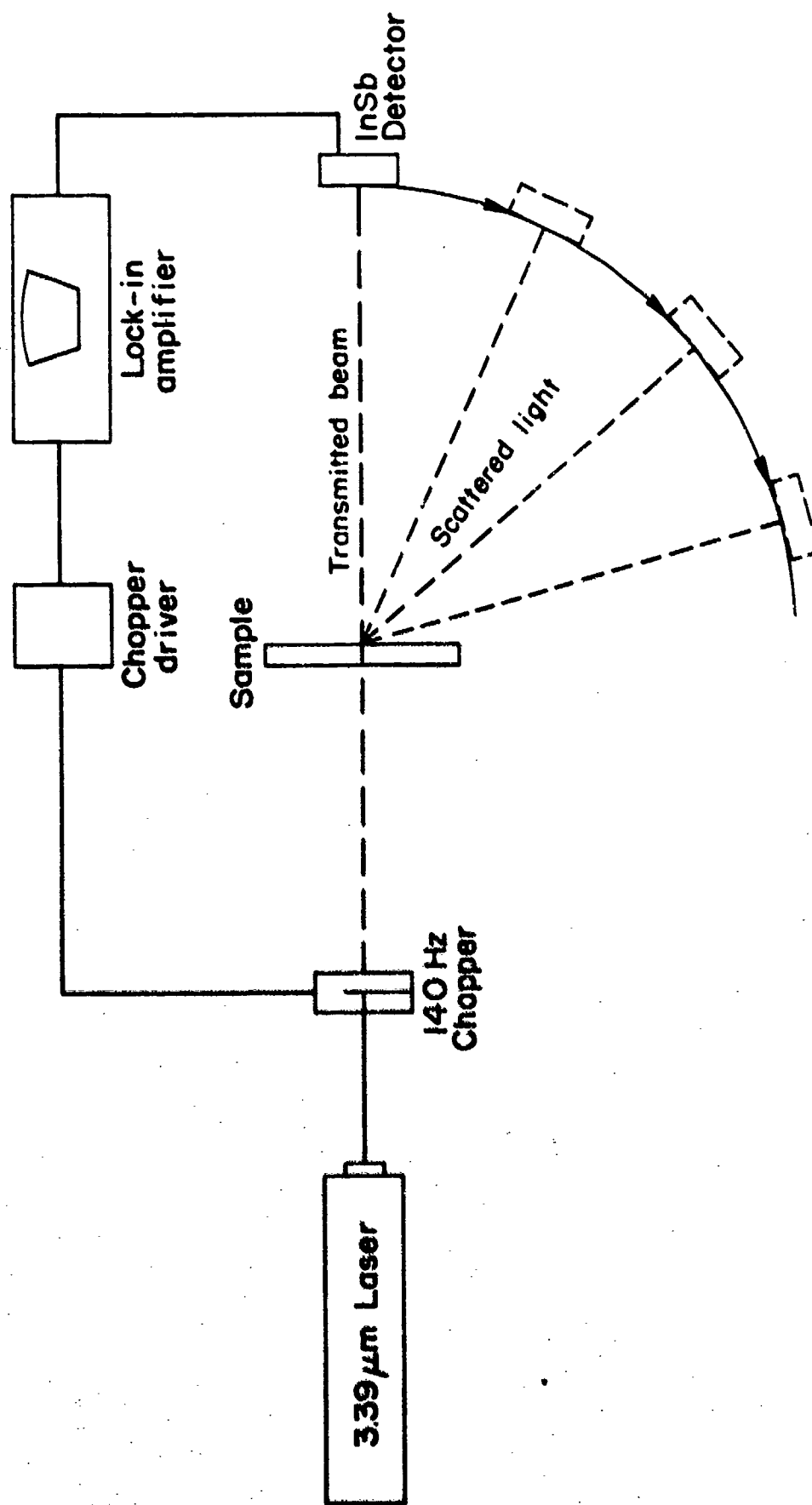
Fig. 10. Schematic of High Angle Scatter Loss Measurement.

3.39 μm Laser

140 Hz Chopper

Chopper driver

Lock-in amplifier

Sample

Transmitted beam

Scattered light

InSb Detector

No sample in-line intensity value ≈ 100,000

○ ZS-70 (Poor visible imaging quality)

● ZS-74 (Good visible imaging quality)

✕ ZnSe (Raytheon CVD)

Scatter intensity per unit thickness of sample (arbitrary units)
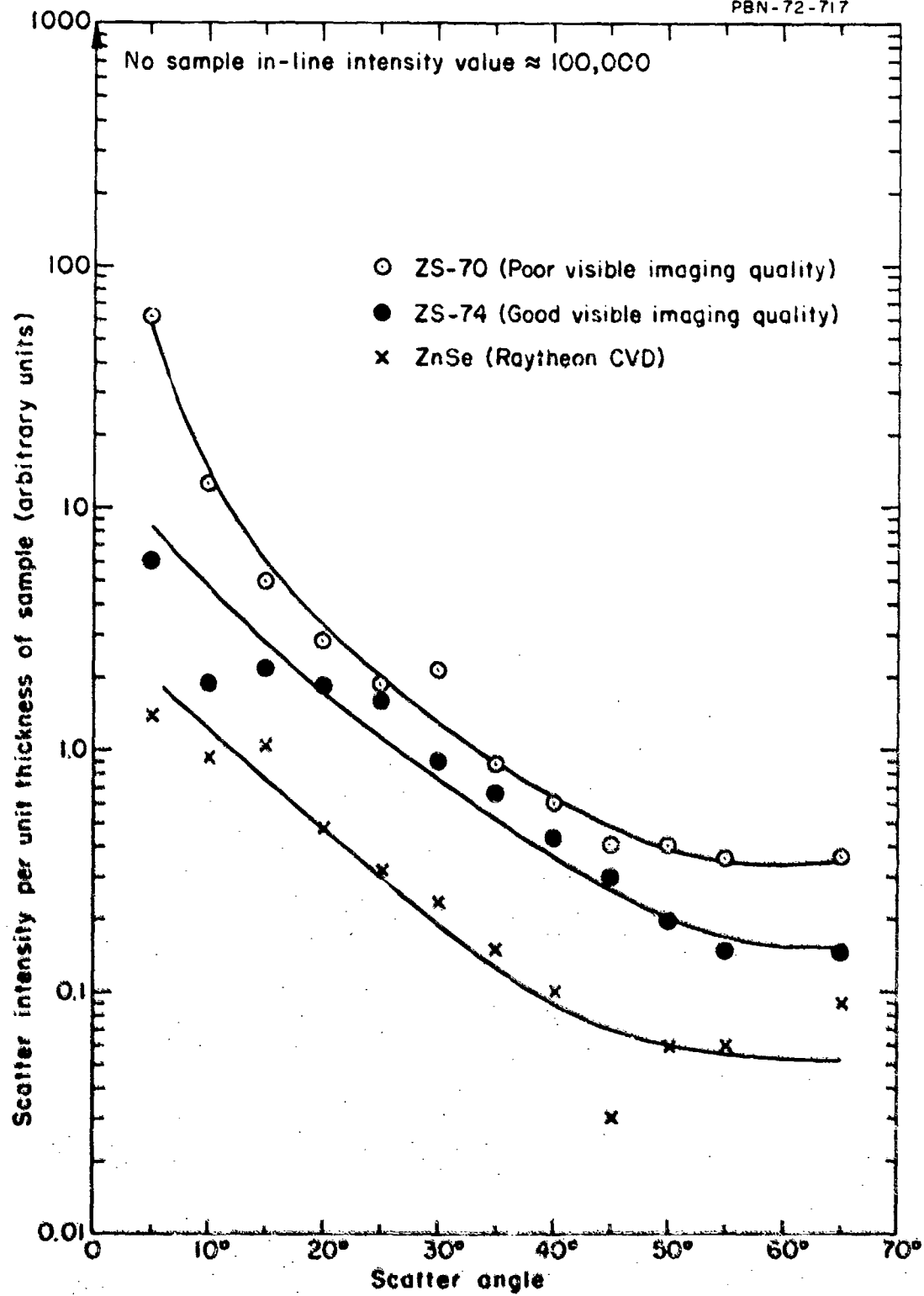
Scatter angle

Fig. 11.   High Angle Scatter Loss for CVD ZnS.

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Runs ZnS-45, ZnS-39, ZS-91, and ZS-93 were chosen for this study. ZnS-45 (550° C deposit had a large band at 6 μm with low scatter, while run ZnS-39 (750° C deposit) had no 6 μm absorption, but a large amount of scatter. Samples, 0.75 X 0.38 X 0.040 in., from each of these runs were cut and polished. The in-line transmission from 2 - 40 μm was measured on each sample prior to annealing. The samples were then either sealed in 4 in. long quartz tubes with the proper atmosphere or put in 4 in. long quartz tubes with one end left open to the desired atmosphere. The annealing schedules used were as outlined in Table IV.

After the anneals the in-line transmission of each sample was remeasured with the as-annealed surfaces and subsequent to a repolish of the surfaces. Several observations can be made from these studies. Annealing sample ZS-39 (high scatter material) under any conditions does not alter the transmission or scatter significantly. Under some annealing conditions, i.e., Zn atmosphere and air @ 75° C, the scattering is increased. The 6 μm absorption band was markedly reduced under all annealing conditions for sample ZS-45. In vacuum and in a zinc atmosphere, it was reduced by a factor of two, and in sulfur and air anneals it was completely eliminated, althougha small absorption appeared at ~ 4 - 5 μm.

The sulfur anneal at 500° C for sample ZS-45 (anneal run #2) did not grossly effect the imaging quality of the material while completely eliminating the 6 μm absorption. This type of anneal appeared to be a feasible means of achieving an extremely desirous material, and lead to additional sulfur annealing experimentation (runs # 4 and 5). In these runs dramatic improvement was observed in the materials as shown in Fig. 12.

4.0 X-RAY ANALYSIS

In an attempt to gain a further insight into the structure of the

TABLE IV

ATMOSPHERE ANNEALING RUNS

Anneal Run	Temp (°C)	Time (hrs)	Running Vac.	Vac.	Zn	Sulfur	Cu	Air	H₂S
No. 1	750	24	---	ZS-39 ZS-45	ZS-39 ZS-45	ZS-39 ZS-45	ZS-39 ZS-45	ZS-39 ZS-45	--- ---
No. 2	500	125	---	ZS-39 ZS-45	--- ---	ZS-39 ZS-45	--- ---	ZS-39 ZS-45	--- ---
No. 3	650	87	ZS-45	---	---	---	---	---	---
No. 4			---	ZS-45 (pps = 760 torr) ZS-45 (pps = 76 torr) ZS-45 (pps = 8 torr)	---	---	---	---	---
No. 5			---	--- ---	--- ---	--- ---	--- ---	--- ---	ZS-91 ZS-93
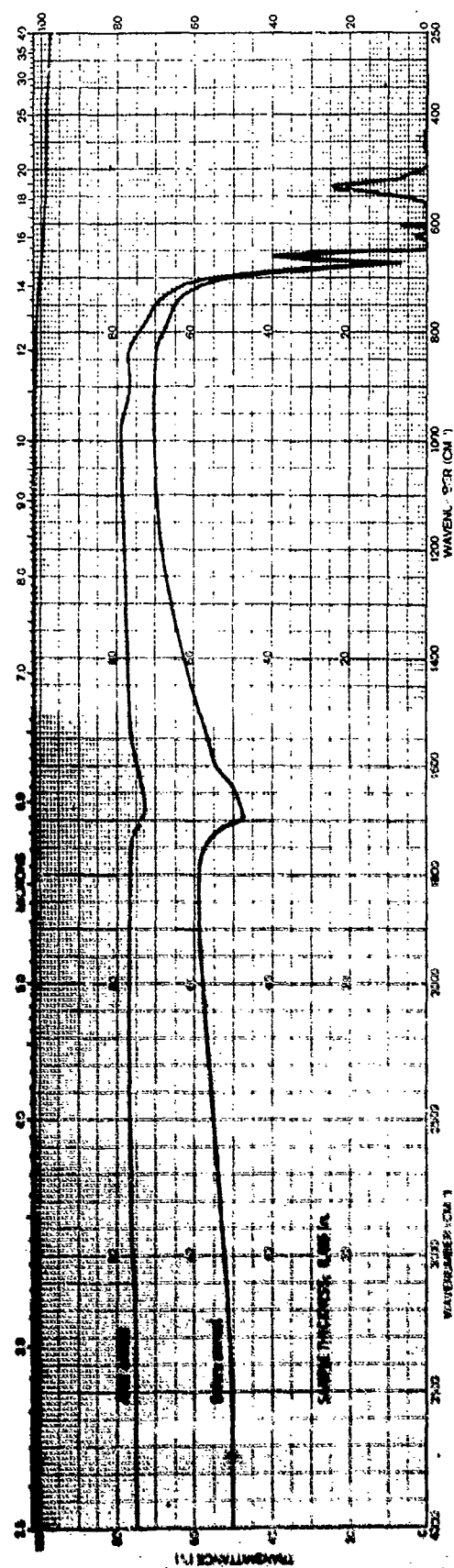
Fig. 12. Improvement of Optical Properties of CVD ZnS by Sulfur Anneal.

CVD ZnS being deposited a limited X-ray investigation was undertaken. Several ZnS materials were investigated by X-ray diffraction using both as-grown deposits and powdered samples. The measurements were made to find out, 1) whether the predominantly cubic deposits contain some small amount of hexagonal phase, and 2) whether the observed diffraction line broadening is due to particle size or strain broadening, or both. The three samples investigated were ZnS-45, -39 and -38. The deposition temperature of these materials are 550°, 750°, and 650° C respectively.

Diffraction traces of the samples ZnS-45, -39, -38, and of a solid solution ZnS-ZnSe suggested the presence of a small background intensity variation in about the position where the hexagonal < 100> peak would occur. On ZnS-38 and ZnS-ZnSe the region of interest was point counted for higher accuracy. This brought out a definite peak of asymmetric shape at the exact hexagonal < 100> position. The asymmetric shape suggests a < 10> band rather than a crystalline < 100 > peak, however. The band would be expected in heavily layer-disordered structures. This interpretation is also supported by the absence of crystalline hexagonal < 101 > peak and by the occurrence of a broad background modulation in its place. Hence, we conclude that the ZnS deposited investigated contain some small amount of hexagonal (wurtzite) phase in strongly layer-disordered form.

Diffraction peak broadening was measured with CuKα radiation on powder samples of ZnS-45 and ZnS-39 as well as on a silicon standard to establish the instrumental broadening. The ZnS linewidths were corrected for instrumental broadening using a Gaussian peak-shape approximation. The corrected widths yielded the following particle sizes:

| | | Particle Size (Å) | |
hkl	2 θ	ZnS-45	ZnS-39
311	56°	267	405
422	88.5°	222	585
531	114.5°	228	615

Allowing for a sizeable scatter in the results due to the rough approximation made in correcting for instrumental broadening we find that the scatter is random and does not exhibit a strong trend toward smaller values with increasing diffraction angle. This suggests predominantly particle size broadening and little if any strain broadening. In X-ray analysis the term, "particle size," cannot be equated with physical particle size, however. Its meaning is that of an average domain size over which the X-rays are scattered coherently. Minor lattice disturbances within a physical particle such as stacking faults, twin boundaries, or dislocation pileups already may constitute boundaries of the coherent domains. The X-ray particle size is therefore more often an indication of the density of lattice defects within a physical particle rather than of its actual size.

5.0 MECHANICAL PROPERTIES

The flexural strength of CVD zinc sulfide was measured for material deposited at temperatures of 750°, 650°, and 550° C. These results are shown in Table V. The flexural strength was measured by 3-point loading on an Instron testing apparatus. Average values of 15,000 psi and higher for the low temperature deposited material encouraged us to further develop this material for FLIR applications. The grain size of the stronger material is very small as would be expected for low temperature material and explains the higher strengths.

6.0 SPECTROGRAPHIC ANALYSIS

Representative samples of CVD ZnS were spectrographically

TABLE V

FLEXURAL STRENGTH OF CVD ZnS

Run No.	Dep. Temp (° C)	Sample No.		Strength (psi)
ZS-39	750	1		9,400
		2		10,200
		3		8,900
		4		10,600
			Avg.	9,800
ZS-38	650	1		15,600
		2		16,000
		3		14,800
		4		14,500
			Avg.	15,200
ZS-45	550	1		21,700
		2		19,000
		3		15,900
		4		12,900
		5		13,400
		6		18,000
			Avg.	16,800

analyzed to determine the impurity content as well as the appearance or
disappearance of impurities as a function of process conditions. Analysis
of material deposited at various temperatures are shown in Table VI.
It is clear from this data that the impurity content of CVD zinc sulfide
is quite low and no significant increase or decrease in these impurities
is found as a function of process conditions evaluated. One important
impurity which could not be evaluated by this technique is oxygen.

JARRELL-ASH DIVISION/FISHER SCIENTIFIC COMPANY
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TABLE VI

Certificate of Analysis

TO: Raytheon Company

28 Seyon Street

Waltham, Mass. 02154

Attn.: Mr. B. DiBenedette, Mr. Swift

DATE REPORTED: 11/5/71

DATE RECEIVED: 11/2/71

ORDER NO. 90-69145

SAMPLE DESCRIPTION: Zn Sulfide

	1	2	3		1	2	3		1	2	3		1	2	3
Li	ND	ND	ND	Zn	H	H	H	Sb	ND	ND	ND	Lu			
Be	ND	ND	ND	Ga	ND	ND	ND	Te	ND	ND	ND	Hf	ND	ND	ND
B	ND	.5	ND	Ge	ND	ND	ND	Cs	ND	ND	ND	Ta	ND	ND	ND
Na	ND	ND	ND	As	ND	ND	ND	Ba	ND	ND	ND	W	ND	ND	ND
Mg	.5	1	.01	Rb	ND	ND	ND	La				Re	ND	ND	ND
Al	.5	.3	.1	Sr	ND	ND	ND	Ce				Os	ND	ND	ND
Si	.3	.5	.1	Y				Pr				Ir	ND	ND	ND
K	ND	ND	ND	Zr	ND	ND	ND	Nd				Pt	ND	ND	ND
Ca	ND	ND	ND	Nb	ND	ND	ND	Sm				Au	ND	ND	ND
Ti	ND	ND	ND	Mo	ND	ND	ND	Eu				Hg	ND	ND	ND
V	ND	ND	ND	Ru	ND	ND	ND	Gd				Tl	ND	ND	ND
Cr	ND	ND	ND	Rh	ND	ND	ND	Tb				Pb	ND	ND	ND
Mn	ND	ND	ND	Pd	ND	ND	ND	Dy				Bi	ND	ND	ND
Fe	.5	.5	ND	Ag	ND	ND	ND	Ho				Th			
Co	ND	ND	ND	Cd	ND	ND	ND	Er				U			
Ni	ND	ND	ND	In	ND	ND	ND	Tm				P	ND	ND	ND
Cu	.1	.1	ND	Sn	ND	ND	ND	Yb							

REMARKS:
1 = ZnS-38
2 = ZnS-39
3 = ZnS-40

Results in ppm

KEY:

ND - Not Detected		T	.01 - .1%
VVFT	< .0001%	L	.1 - 1%
VFT	.0001% - .001%	M	1% - 10%
FT	.001% - .01%	H	>10%

STAFF ANALYST

SUPERVISOR,
TESTING LABS

40

TL 21-103

JARRELL-ASH DIVISION/FISHER SCIENTIFIC COMPANY
590 LINCOLN STREET, AT ROUTE 128 WALTHAM, MASSACHUSETTS 02154 USA

Table VI (Cont'd)

Certificate of Analysis

TO: _____Raytheon Company_____

_____28 Seyon Street_____

_____Waltham, Mass. 02154_____

Attn.: Mr. B. DiBenedette, Mr. Swift

DATE REPORTED: _11/5/71_

DATE RECEIVED: _11/2/71_

ORDER NO. _90-69145_

SAMPLE DESCRIPTION _____Zn Sulfide_____

	4	5			4	5			4	5			4	5	
Li	ND	ND		Zn	H	H		Sb	ND	ND		Lu			
Be	ND	ND		Ga	ND	ND		Te	ND	ND		Hf	ND	ND	
B	ND	ND		Ge	ND	ND		Cs	ND	ND		Ta	ND	ND	
Na	ND	ND		As	ND	ND		Ba	ND	ND		W	ND	ND	
Mg	.5	.02		Rb	ND	ND		La				Re	ND	ND	
Al	.1	.5		Sr	ND	ND		Ce				Os	ND	ND	
Si	.1	.1		Y				Pr				Ir	ND	ND	
K	ND	ND		Zr	ND	ND		Nd				Pt	ND	ND	
Ca	ND	ND		Nb	ND	ND		Sm				Au	ND	ND	
Ti	ND	ND		Mo	ND	ND		Eu				Hg	ND	ND	
V	ND	ND		Ru	ND	ND		Gd				Tl	ND	ND	
Cr	ND	ND		Rh	ND	ND		Tb				Pb	ND	.5	
Mn	ND	ND		Pd	ND	ND		Dy				Bi	ND	ND	
Fe	ND	ND		Ag	ND	ND		Ho				Th			
Co	ND	ND		Cd	ND	ND		Er				U			
Ni	ND	1		In	ND	ND		Tm				P	ND	ND	
Cu	.5	.3		Sn	ND	ND		Yb							

REMARKS: _____4 = ZnS-44_____

_____5 = ZnS-45_____

_____Results in ppm_____

KEY:

ND – Not Detected		T	.01 –.1%
VVFT < .0001%		L	.1–1%
VFT .0001% – .001%		M	1% –10%
FT .001% – .01%		H	>10%

signature
STAFF ANALYST

signature
**SUPERVISOR,
TESTING LABS**

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SECTION VI
CONCLUSIONS

The feasibility of utilizing the chemical vapor deposition process to fabricate large aperture infrared windows of zinc sulfide was demonstrated by making plates of this material whose nominal size was 14 × 22 × 0.45 in. The optical imaging quality, strength, and in-line transmission of material from these plates is significantly better than that fabricated during the previous program. The process was modified to the extent that the optical imaging quality of the material, particularly in the visible, was significantly improved. This was accomplished by determining a more optimum deposition temperature ($\sim 650°$ C) and by using a $H_2S/$ Zn molar ratio of approximately 0.5.

Of the various mandrel materials investigated the best results were attained with graphite coated with a thin layer of colloidal graphite. The same mandrel can be used repeatedly. A few runs were also made on optically polished fused silica. The texture of the ZnS deposited on this material is significantly better than that deposited on graphite. However, because of the initial expense of a large fused silica mandrel the use of this material was not pursued.

Annealing ZnS samples deposited between 500° and 700° C in various atmospheres eliminates the absorption band at 6 μm. Preliminary results indicate that a sulfur anneal at 500° C is most beneficial. The material color is altered from yellowish-brown to colorless. The imaging resolution of this material in thin sections (~ 0.050 in) is considered to be excellent.

The problems encountered in the chemical vapor deposition process are quite different than those encountered in hot pressing or glass casting. Impurity absorptions which cause difficulties in these latter two

processes were of second-order importance for zinc sulfide fabricated by the CVD process. The only absorption observed in an infrared scan was at $\sim 6 \mu$m, whose cause at present is not completely understood. There are, of course, unique problems that are peculiar to the chemical vapor deposition process. Among these are the formation of nodules, the inclusion of zinc in the large plates, and low angle scattering, particularly in the visible.

SECTION VII
RECOMMENDATIONS

It has been demonstrated that the chemical vapor deposition pro-
cess is not inherently size limited and that large plates ($\sim 14 \times 22$ in.)
can be fabricated by this process. This potential should be further
exploited by improving the optical quality of the material, particularly
in the visible. This improvement can be accomplished by the following
experiments:

1) Redesign the exhaust end of the mandrel and the chemical
vapor deposition furnace to eliminate zinc inclusions.

2) Improve the thickness distribution of the large plates ($14 \times$
22 in.) (transversely and axially) by reducing turbulence.

3) Further improve the uniformity of the material in the thick-
ness direction by controlling the usage rate of the zinc compound more
closely as a function of time during the run.

4) Investigate further the use of post-deposition annealing as a
means of improving optical properties.

5) Determine the cause of scattering sites in the visible and de-
vise means of eliminating them.

6) Extend the useful range over which zinc sulfide is operative
by forming solid solutions with cadmium sulfide and/or zinc selenide.

7) Fabricate complex shapes such as hemispherical-end
cylinders.

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DOCUMENT CONTROL DATA - R & D

(Security classification of title, body of abstract and indexing annotation must be entered when the overall report is classified)

1. ORIGINATING ACTIVITY (Corporate author)	2a. REPORT SECURITY CLASSIFICATION
Raytheon Research Division Waltham, Massachusetts 02154	Unclassified
	2b. GROUP N/A

3. REPORT TITLE

Chemical Vapor Deposition of IR Materials for Reconnaissance

4. DESCRIPTIVE NOTES (Type of report and inclusive dates)

Final Technical Report -- June 15, 1971 to June 15, 1972

5. AUTHOR(S) (First name, middle initial, last name)

Bernard A. diBenedetto, James Pappis, Anthony J. Capriulo

6. REPORT DATE	7a. TOTAL NO. OF PAGES	7b. NO. OF REFS
September 1972	44	0

8a. CONTRACT OR GRANT NO. F33615-71-C-1775 b. PROJECT NO. 6102 c. Task No. 01 d.	9a. ORIGINATOR'S REPORT NUMBER(S) S-1468 9b. OTHER REPORT NO(S) (Any other numbers that may be assigned this report) AFAL-TR-72-309

10. DISTRIBUTION STATEMENT

Distribution limited to U. S. Government agencies only: report contains test and evaluation information; September 1972. Other requests for this document must be referred to AFAL/TEL, Wright-Patterson AFB, Ohio.

11. SUPPLEMENTARY NOTES	12. SPONSORING MILITARY ACTIVITY
N/A	Air Force Avionics Laboratory Wright-Patterson AFB, Ohio 45433

13. ABSTRACT

The significance of this research and development program to the Air Force is the demonstrated feasibility of fabricating large infrared transmitting windows of zinc sulfide with good physical characteristics by the chemical vapor deposition (CVD process. Plates approximately 1 ft. × 2 ft. with good transmission between 8 and 12 μm were made and delivered as window blanks. Furthermore, the imaging properties of CVD ZnS, particularly in the visible end of the spectrum were significantly improved in comparison to material fabricated during an earlier program. It was also shown that it may be possible to extend the useful transmission range of zinc sulfide by fabricating a solid solution with zinc selenide.

Material fabricated during this program had a transmittance in excess of 60 percent from 6 to 12.5 μm, a flexural strength of 15,000 psi, and a use limit above 200° C. The potential for improving the optical quality of the material through better control of the process and/or annealing the material after deposition was also demonstrated.

DD FORM 1473

14. KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
Infrared windows						
Optical transmission						
Optical scatter						
Zinc sulfide						
Zinc sulfide-zinc selenide						